PATENT SPECIFICATION

922,619

DRAWINGS ATTACHED.

Date of Application and filing Complete Specification :
Nov. 4, 1960. *No. 38005/60.*

Application made in South Africa (No. 4393) on Nov. 5, 1959.

Complete Specification Published : April 3, 1963.

Index at Acceptance :—Class 1(1), F19.

International Classification :—B01j.

COMPLETE SPECIFICATION.

ERRATUM

SPECIFICATION NO. 922,619

Page 1, line 12, *for* "Johannesburgh" *read* "Johannesburg"

THE PATENT OFFICE,
23rd May 1963.

DS 74249/1(6)/R.109 200 5/63 PL

SPECIFICATION NO. 922,619

By a direction given under Section 17 (1) of the Patents Act 1949 this application proceeded in the name of ADAMANT LABORATORIES (PROPRIETARY) LIMITED, of 45, Main Street, Johannesburg, Republic of South Africa, a body corporate organised according to the laws of the Republic of South Africa.

THE PATENT OFFICE

D 33164/1 200

...told, the purpose for which the die of the invention has been evolved is to compress the charge and heat it to elevated temperature simultaneously, so that the object of the invention is also to provide a die which can succesfuly be applied to this end.

The production of dies which are capable of imposing on the material charged into them high pressures of the order of 50,000 atmospheres and above presents no great technical difficulties. Indeed, in laboratory equipment pressures up to 80,000 atmospheres have been achieved. Similarly, there is no difficulty in bringing material to elevated temperature, say up to 1800° C. and this can readily be achieved while the material is kept under moderate pressure. Where, however, grave difficulties do arise is when material is to be heated to these temperatures and simul-

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The detailed construction of the preferred form of the die will best be understood if it be described with reference to the accompanying drawings. In these drawings:

Figure 1 is a vertical section;

Figure 2 is a section of the pressure zone on an enlarged scale;

Figure 3 is a section through the assembly within the pressure chamber;

Figure 4 is a modification of Figure 3;

Figure 5 is a detail view of another form of charge;

Figure 6 is a detail view of a modification of the arrangement of Figure 5; and

Figure 7 is a detail view of a modified core.

In the drawings, the core is a ring 10 which is cylindrical and dished on both its faces. The anvils 12, 14 are also cylindrical and are formed with opposed frusto-conical or -con-

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COMPLETE SPECIFICATION.

A New High-Pressure Die.

We, JAN FRANS HENRI CUSTERS, a Subject of the Queen of the Netherlands, of 18 Finger Street, Cyrildene, Johannesburg, South Africa, HENRY BROOKE DYER, a Citizen of the Union of South Africa, of 1504 George Street, Bryanston, Johannesburg, South Africa, BERNARD WILFRED SENIOR, a British Subject, of 1927 Devonshire Avenue, Bryanston, Johannesburg, South Africa, and PETER THEO WEDEPOHL, a Citizen of the Union of South Africa, of 4 Roseneath Mansions, Hillbrow, Johannesburgh, South Africa, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement :—

The object of the present invention is to provide a die capable of exerting pressure on material enclosed within its pressure chamber of the order of say 50,000 atmospheres and above. While it is not excluded that the die be used to compress the material in the cold, the purpose for which the die of the invention has been evolved is to compress the charge and heat it to elevated temperature simultaneously, so that the object of the invention is also to provide a die which can successfuly be applied to this end.

The production of dies which are capable of imposing on the material charged into them high pressures of the order of 50,000 atmospheres and above presents no great technical difficulties. Indeed, in laboratory equipment pressures up to 80,000 atmospheres have been achieved. Similarly, there is no difficulty in bringing material to elevated temperature, say up to 1800° C. and this can readily be achieved while the material is kept under moderate pressure. Where, however, grave difficulties do arise is when material is to be heated to these temperatures and simultaneously maintained under high pressure of say 80,000 atmospheres.

In its broadest aspect the die of the invention consists in an annular core with a dished face, an anvil having a frusto-conical or -conoidal peak that is accommodated within the dishing of the core, is separated from the core by empty space, and occludes the adjacent end of the cavity in the core; means occluding the other end of the cavity; and a hollow body within the cavity that, with the anvil and the means occluding the other end of the cavity in the core, forms a pressure chamber, the body being made of a refractory material characterized by low compressive strength and high internal friction.

In the preferred form, the die has two peaked anvils accommodated within a core dished at opposite faces.

The core may have a central insert of expandable material characterized by high resistance to compressive forces; or it may be integral.

The detailed construction of the preferred form of the die will best be understood if it be described with reference to the accompanying drawings. In these drawings:

Figure 1 is a vertical section;

Figure 2 is a section of the pressure zone on an enlarged scale;

Figure 3 is a section through the assembly within the pressure chamber;

Figure 4 is a modification of Figure 3;

Figure 5 is a detail view of another form of charge;

Figure 6 is a detail view of a modification of the arrangement of Figure 5; and

Figure 7 is a detail view of a modified core.

In the drawings, the core is a ring 10 which is cylindrical and dished on both its faces. The anvils 12, 14 are also cylindrical and are formed with opposed frusto-conical or -con-

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oidal peaks 16, 18 that are accommodated within the depressions in the ring 10 that separates the anvils, so that the cavity of the ring is closed off by the flattened ends of the peaks to define a pressure chamber 20. The corners of the ring directed towards the chamber 20 are radiused, while the corners 22 of the peaks are sharp.

The included angle of the peaks may be somewhat less than the angularity of the dishing of the ring, so that the opposed faces 24, 26 and 28, 30 of the ring and the anvils diverge outwardly at an angle of about two degrees; or the opposed faces may be parallel.

The ring and the anvils are made of refractory material such as cemented hard-metal. The properties of the hard-metal are selected to give maximum compressive strength to the anvils and good compressive strength allied with high tensile resistance to the ring. For the anvils a fine to medium grained tungsten carbide with a low content of cobalt may be used. For the ring, a medium to coarse grained tungsten carbide with medium cobalt content is suitable.

The ring 10 is buttressed against bursting forces by a ring or rings of high-tensile, steel. In the drawings, three rings, 32, 34 36 are shown. These are shrunk or forced-fitted on to the ring 10. The anvils are buttressed each by a ring 38 or 40 of high tensile steel, also shrunk or force-fitted on to the anvils.

If it be required that the die be liquid-cooled, then there are additional rings 42, 44, 46 around the two anvils and the ring 10 respectively. Between the ring 10 and each anvil there are located two rubber or rubber-like toroidal rings 48, 50; 52, 54 that provide between them annular spaces 56, 58. Each pair of totoidal rings 48, 50; 52, 54 may be mounted on a thin annular plate 60, 62 to assist in locating the toroidal rings within the assembly.

Fluid is injected into the spaces 56, 58 through dog-legged ducts 64, 66 in the rings 42, 44, and withdrawn through similar ducts 68, 70 diametrically opposed to the ducts 64, 66.

The assembly is held together gravitationally with the ring 10 and its surrounding supporting rings spaced from the anvils and their supporting rings, (and with the opposed flat, transverse faces parallel) by the toroidal rings 48, 50, 52, 54. If the die need not be liquid cooled, only one toroidal ring, to act as a spacer, will be necessary between the ring 10 and each anvil. In this case the outer rings 42, 44, 46 are unnecessary.

In a modified form of core (Figure 7) the central part is constituted by an insert formation 72 of refractory material, a sintered aluminium oxide. The material must be characterized by high resistance to compressive forces. The advantage of this construction is that after use the insert, which will inevitably suffer damage, can be removed and replaced by a fresh insert. The outer part of the core is little likely to suffer serious damage, at least during several uses, and can therefore be reused, thereby economizing on cost.

In the operation of the die, the pressure chamber 20 is filled with a soft, compressible, porous, thermally and electrically insulating body 74, possessing low compressive strength and high frictional properties, and hollowed at 76 to receive the charge 77 (Figure 2). A suitable material is the natural stone known as pyrophyllite or wonderstone. The quantity of this material is specific and highly critical and small variations in this quantity give rise to different pressures in the chamber 20 with respect to a given load between the anvils 12, 14. The initial shape of the body has been shown to affect the recorded pressure with relation to load only slightly and many shapes are possible to suit particular circumstances. A cylinder with an indented periphery 78 (Fig. 3) has been found completely satisfactory.

In operation the assembly, with a charge within the pressure chamber 20, is placed between the platens of a press and load is exerted through the anvils 12, 14 on to the charge in the chamber 20. Accurate alignment of the anvil and ring 10 and its supporting rings is necessary. Circumferential alignment may be obtained by the use of a straight-edge along the periphery of the assembly, or by a clamp or flexible belt embracing the whole assembly. Horizontal parallelism is looked after by the rings 48, 50; 52, 54.

On applying pressure, the body 74 is compressed (Figure 2), and as the pressure increases the material tends to flow into the gaps 80, 82 between the anvils and the die ring 10. As the pressure further increases, these gaps are reduced more and more by the inward movement of the anvil faces in consequence of which the body 74 and the charge in the chamber 20 become compressed more and more. The outward flow of material through the gaps 80, 82 is inhibited by friction on the die and anvil faces. The conformation of each gap 80, 82 is such that the gap progressively narrows in the outward direction, towards the throats 84, 86, which apart from the closing movement of the die, itself inhibits extrusion of the material of the body into the gaps 80, 82.

Thus, the body 74 itself provides a barrier that is sufficient to contain the mounting pressures without the necessity for washers, seals or gaskets between the adjacent surfaces of the core and the anvils.

Further assistance may be given by coating the operative faces of the anvil and the ring 10 with a high friction abrasive material such

as jeweller's rouge. Also the material of the body 74 may be impregnated with a suspension of rouge to increase the friction.

By suitable design of the shape of the anvils 12, 14 and the ring 10 a flow of the material of the body 74 may be obtained which is compatible with the maintaining of a particular internal pressure within the pressure chamber. With the particular die configuration shown in Figures 1 and 2 pressures of 70,000 atmospheres may be maintained by extrusion of material into the gap 80 of about 0.020" by 0.75" mean diameter, measured normal to the opposed faces defining the gaps.

So much for the production of pressure within the pressure chamber 20. The matter of heating will now be considered.

Heat is imparted to the charge within the pressure chamber by electrical current flow through a resistance element.

It is here necessary to consider the nature of the charge. If it be an electrical conductor, as it is assumed to be in Figures 2, 5 and 6, then the charge is itself the resistance element. Electrical current is fed along a path comprising the anvils 12, 14 and the charge. In Figures 2, 5 and 6, the path includes also two steel rings 88, 90 interposed between the anvils and the charge 77 and non-conductive plugs 92, 94 within the cavities of the rings 88, 90. The purpose of these rings is to prevent direct contact between the charge and the faces of the anvils, to prevent damage to them by the compressed and heated charge. The plugs 92, 94 may be of pyrophyllite.

In Figure 6, the rings 88, 90 are formed lateral flanges 96, against which the anvils press.

If, on the contrary, the charge be a non-conductor or a poor conductor of electricity, as is contemplated in Figures 3 and 4, then the circuit must be completed by a resistance element other than the charge. In Figure 3 this element is shown as a tube 100. It is located within the cavity of the body 74. The most suitable materials for the tube 100 are tungsten, molybdenum or graphite but other metals or electrically conducting non-metals may be used. The ends of the heating tube 100 are sealed by electrically conducting pads 102 making good electrical contact with the anvil faces and giving a relatively low resistance and therefore low heat dissipation at the anvil surfaces. The pads 102 may be of the same material as the tube 100 and graphite is generally preferred for both because of its convenience for machining to shape. The faces of the anvils may be covered with metal shims 104 to improve electrical contact and to prevent diffusion of the material of the heating tube 100 and end pads 102 into the refractory carbide of the anvils, thus impairing their mechanical properties. This has been found to be particularly important where a graphite heating tube and end pads are used.

Inside the conducting pads 102 may be fitted insulating pads 106 either for the purpose of inhibiting axial heat flow which would mechanically weaken the anvil faces, or for the purpose of shielding the charge from the electric current. Also an inner tube 108 of electrically insulating material may be used. Both the pads 106 and the tube 108 serve the additional purpose of separating the charge 77 in the pressure chamber 20 defined by the pads 106 and the tube 108 from the material of the heating tube 100 and end pads 102 in cases where it would react chemically with them or diffuse through them. Suitable materials for the pads 106 and the tube 108 are refractory oxides such as alumina, zirconia and silica in fused or crystalline form or mineral insulators such as wonderstone.

The heating current is passed through the anvils 12 and 14 via the pads 102 and the tube 100. The resistance of the tube 100 is much greater than that of the anvils and other parts of the electrical path, causing the major part of the heat to be generated in the pressure chamber 20. Where an internal axial temperature gradient is required, this may be procured by using a heating tube which has a continuously or discontinuously varying cross-sectional area.

The heating tube 100 may be replaced by one or more rods 110, as shown in Figure 4, which function in the same way as the tube and may be of the same material.

As a general comment, it may be said that the body 74 could consist in an outer part of high-friction material such as pyrophyllite to minimize extrusion into the gaps between the anvils and the core, and an inner part of material such as talc or silver chloride incapable of supporting high shear stress. This could assist in minimizing pressure gradients within the charge.

In the case of a conductive or a non-conductive charge, alternating or direct current may be used.

Given an assembly in which the body 74 is of the order of 12mm in diameter, the apparatus described has been found to be capable of generating pressures within the pressure chamber of the order of 80,000 atmospheres, while the charge is subjected to temperatures of the order of 2000° C.

WHAT WE CLAIM IS:—

1. A high pressure die consisting in an annular core with a dished face, an anvil having a frusto-conical or -conoidal peak that is accommodated within the dishing of the core, is separated from the core by empty space, and occludes the adjacent end of the cavity in the core; means occluding the other end of the cavity; and a hollow body within the cavity that, with the anvil and the means occluding the other end of the cavity in the

core, forms a pressure chamber, the body being made of a refractory material characterized by low compressive strength and high internal friction.

2. The die of Claim 1 in which the core is dished on both faces and the means occluding the other end of the cavity consists in a second anvil identical with the first anvil.

3. The die of Claim 1 or Claim 2 in which the gap between the core and the or each anvil diverges outwardly.

4. The die of Claim 3 in which the extent of divergence is about two degrees.

5. The die of any of the above claims including a metal shim covering the or each anvil in the zone of the cavity of the core.

6. The die of any of the above claims in which the or each anvil and the core are surrounded by flat annular buttressing rings in a plane perpendicular to the principal axis of the core, the rings being separated by deformable spacing elements.

7. The die of Claim 6 in which the elements are toroidal rings between each pair of annular rings.

8. The die of Claim 7 in which the ring is of an elastic material such as rubber.

9. The die of any of the above claims in which the body that is hollowed to define the pressure chamber is made of pyrophyllite.

10. The die of any of the above claims in which the radially inner part of the core adjacent to the body is constituted by an inserted element of refractory material.

11. The die of any of the above claims including a charge enclosed within the pressure chamber, and means for electrically heating the charge.

12. The die of Claim 11 in which the charge is electrically conductive, and including conductive spacing means between the or each anvil and the charge.

13. The die of Claim 11 as dependent on Claim 2 in which resistance heating means associated with the charge is arranged between the anvils.

14. The die of Claim 13 in which the resistance heating means is a resistance element in the form of a tube containing the charge.

15. The die of Claim 13 in which the resistance heating means is a rod embedded in the charge.

16. The die of any of the above Claims 6 to 8 comprising buttressing rings for the core and the or each anvil defining between them and with the deformable elements a space or spaces, and means to flow cooling fluid through the space or spaces.

17. The die of Claim 16 including ducts in the buttressing rings to lead cooling fluid into and out of the space or spaces.

18. A high-pressure die substantially as herein described with reference to Figure 1 of the accompanying drawings.

19. A high-pressure die substantially as herein described with reference to Figure 3 of the accompanying drawings.

20. A high-pressure die substantially as herein described with reference to Figure 4 of the accompanying drawings.

21. A high-pressure die substantially as herein described with reference to Figure 5 of the accompanying drawings.

22. A high-pressure die substantially as herein described with reference to Figure 6 of the accompanying drawings.

23. A high-pressure die substantially as herein described with reference to Figure 7 of the accompanying drawings.

MARKS & CLERK,
Chartered Patent Agents,
Agents for the Applicants.

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Published at The Patent Office, 25, Southampton Buildings, London, W.C.2,
from which copies may be obtained.

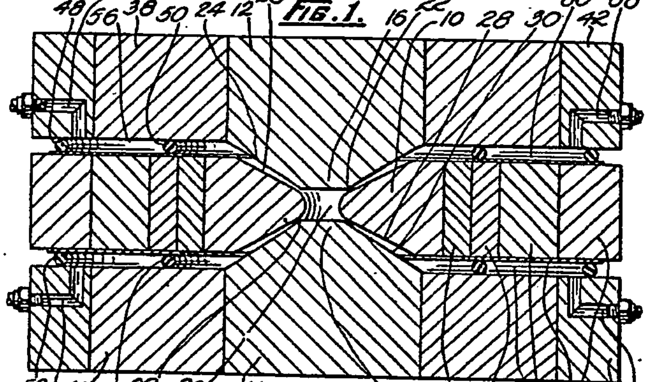


FIG. 1.

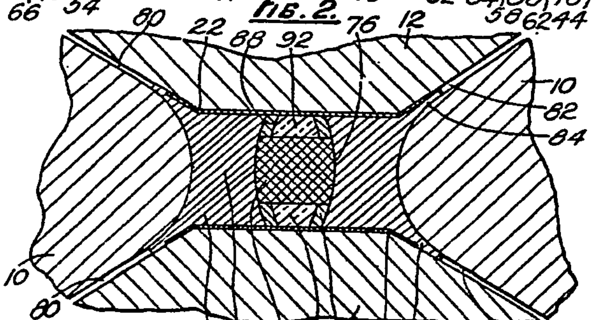


FIG. 2.

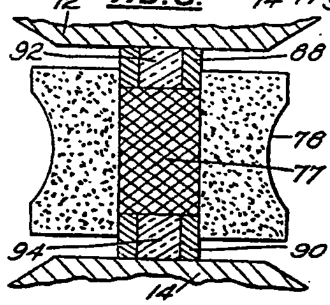


FIG. 5.

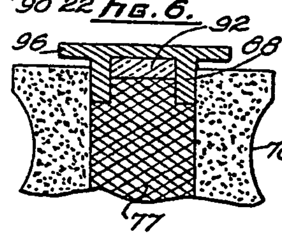
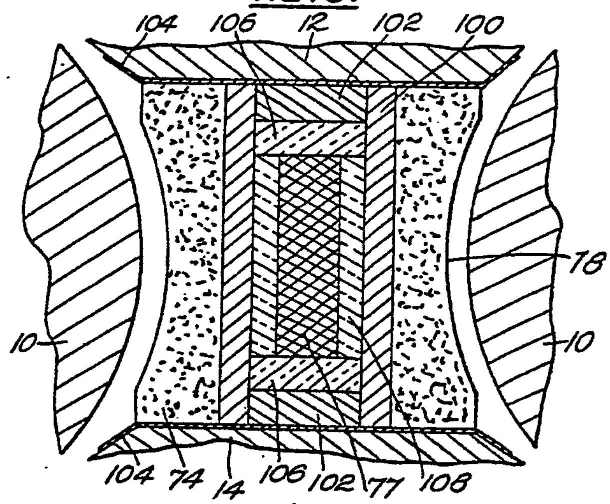


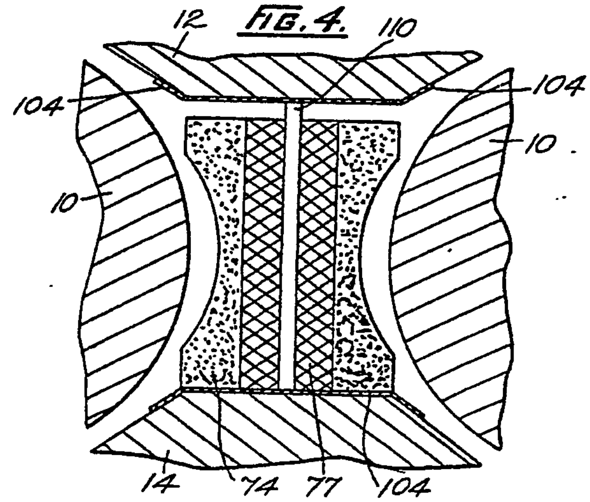
FIG. 6.

FIG. 3.



104
106
12
102
100
78
10
10
104
74
14
106
102
77
108

FIG. 4.

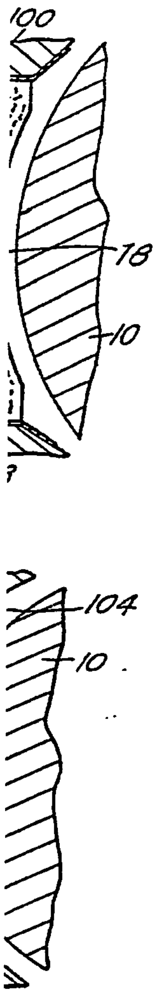


12
110
104
104
10
10
14
74
77
104

922619 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheets 2 & 3



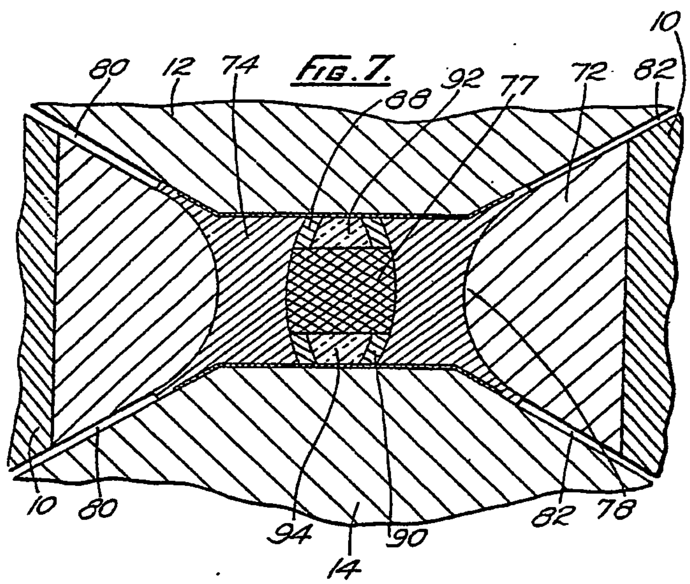


FIG. 7.

922619 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheets 2 & 3

FIG. 3.

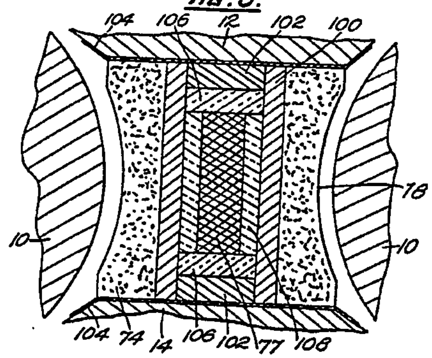


FIG. 4.

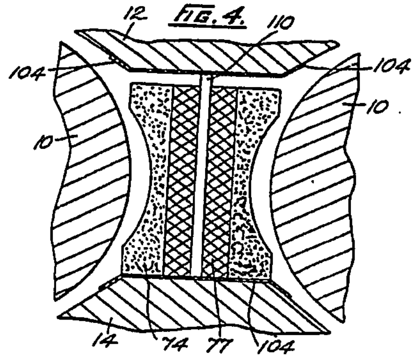


FIG. 7.